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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] It covers with this invention at the time of the powder for ****, or ceramic fabrication sintered-compact baking, and it relates to the manufacture method of a spherical rare-earth-elements oxide useful as an object for powder.
[0002]
[Description of the Prior Art] When it is going to generate ****** of a rare-earth-elements oxide, in order to obtain a film with the uniform direction with the sufficient flow nature of the powder supplied to a burner, a particle with it is liked. [it is convenient, therefore spherical] Moreover, it covers and the spherical particle with which it covers in order to prevent adhesion with internal insulation and a reaction, in case a ceramic Plastic solid is sintered and which a fluidity is good also as powder, and there are few touch areas, and ends is liked.
[0003] Publication number Although the method of corning using a fines-like rare-earth-elements oxide to No. 23214 [three to], and obtaining a spherical particle is indicated, since it manufactures using it once manufacturing a particle by baking a precipitation generation and ** exception from a solution, by this method, it takes time and effort.
[0004]
[Problem(s) to be Solved by the Invention] this invention is what solved the above-mentioned fault, and tends to offer the manufacture method which can obtain easily from solution the object for ****, and the rare-earth-elements oxide spherical particle which covered and was suitable for powder.
[0005]
[Means for Solving the Problem] It is what this invention persons repeated [what] examination in order to solve this technical problem, the manufacture method of of the object for **** and the spherical rare-earth-elements oxide which covered and fitted powder with which productivity consists of a good simple process was found [what] out, and manufacture conditions were established [what], and completed this invention. In case the summary is mixed, makes the solution of the water-soluble salt of rare earth elements, and the solution of ammonia or hydroxylation alkali react and obtains precipitation of a rare-earth-elements hydroxide It is in the manufacture method of the rare-earth-elements hydroxide characterized by making it distribute in the organic solvent which is not mixed with water and homogeneity, and adding at least one of this rare-earth-elements solution and this alkali solution, and the manufacture method of the rare-earth-elements oxide by calcinating it.
[0006] While the spherical particle-like rare-earth-elements oxide was obtained simply and was economical by obtaining a hydroxide from the solution of the water-soluble salt of rare earth elements easily according to the invention in this application, and calcinating this, all consisted of the spherical particle, and the angle of repose was also small, it covered with it at the time of the powder for ****, or ceramic sintering, and it turns out that most of this particle is useful as powder.
[0007] Hereafter, this invention is explained in detail. An yttrium and the atomic number of the adaptation range of this invention are the lanthanoidses of 57-71 as rare earth elements.

[0008] The ratio [as opposed to / being spherical / a true sphere and a minor axis] of a major axis in this invention 1.5 or less particle of a **** globular form is meant. This is sufficient range on a use. Moreover, the oxide which consists of such a particle has the angle of repose (it measures with a gradient method) smaller than the usual oxide which shows fluid goodness. A mean particle diameter (D50) is what was expressed with volume criteria, and 50% of total particle volume is occupied by the particle below a mean particle diameter. The measuring method used the Coulter counter (tradename by the coal tar company).

[0009] In case the solution of the water-soluble salt of rare earth elements and the solution of ammonia or hydroxylation alkali are made to react and precipitation is deposited as the manufacture method of the spherical rare-earth-elements hydroxide of this invention, it adds as the so-called w/o type emulsion which was made to distribute at least one side of rare-earth-elements solution and alkali solution in the organic solvent which is not mixed with water and homogeneity, and **** distributed in the continuous phase of the organic solvent. A nitrate, a chloride, etc. are used as a water-soluble salt of rare earth elements. Since viscosity will go up too much after precipitation generation if not much high [if one sort or two sorts or more are sufficient as the kind of rare earth elements and total rare-earth-elements concentration is not much low, it increases / volume / and is uneconomical, and] again 0.1 - 2.0 mol/l is good.

[0010] When disliking mixing of alkali metal etc. although the solution of ammonia, such as a sodium hydroxide, a potassium hydroxide, and a barium hydroxide, is used in order to settle rare earth elements, it is good to use aqueous ammonia. By the same reason as the place of a rare-earth-elements solution also described this concentration 0.2 - 5.0 mol/l is good. As an amount, it is one mol of rare earth elements. It receives and is 3-5 mols. It is suitable and is three mols. In the following, yield is bad and it is five mols. The effect is [nothing] and is uneconomical even if it exceeds.

[0011] As an organic solvent, be [easy although / it] not mixed with water and homogeneity, the mixture obtained by fractional distillation of petroleum, such as annular aliphatic hydrocarbon, such as straight chain aliphatic hydrocarbon, such as aromatic hydrocarbons, such as toluene, and n-hexane, and a cyclohexane, and kerosine, is mentioned as an example. In consideration of the height of the flash point, the safety to a human body, a price, etc., kerosine, a cyclohexane, etc. are desirable in inside. It is convenient to generation of an w/o emulsion to use one 2 to 5 times the amount of this by volume as an amount to the solution to distribute.

[0012] Moreover, the organic solvent is received in comparatively oil-soluble emulsifiers, such as sorbitan monooleate, polyoxyethylenesorbitan monostearate, and sorbitan trioleate, in this case. It adds 0.5 to 5.0% of the weight.

[0013] It shakes, it mixes with the organic solvent which melted the emulsifier first by churning etc., and the thing which add by making it an emulsion among an alkali solution and a rare-earth-elements solution as the mixed method of a solution is emulsified. It can be checked by adding oil-soluble or water-soluble coloring matter with an optical microscope etc. whether the w/o emulsion has been made well. Although whichever is sufficient as the mixed sequence of the comrades of an emulsion when making both rare-earth-elements solution and an alkaline-water solution into an emulsion, when mixing only in solution, as for one of the two, it is good to add the emulsion to solution. The time which this mixture takes is arbitrary and good. The temperature at the time of mixing a solution has a good room temperature. Even if it makes it low temperature, since the organic solvent is used, it is not desirable on safe for it to be ineffective and to make it an elevated temperature.

[0014] If it finishes adding all solutions, in order to advance a reaction completely, it will set 10 minutes or more. If generation of precipitation is completed, with a Buchner funnel, it will carry out a ** exception and will rinse. The spherical rare-earth-elements hydroxide was obtained now. Obtained hydroxide 600 degrees C or more A spherical rare-earth-elements oxide is obtained by calcinating below 1,500 degrees C.

[0015]

[Example] Hereafter, although an example is given and the embodiment of this invention is explained, this invention is not limited to these.

Example 1 kerosine What added sorbitan monooleate 1.5g to 200ml, and was dissolved was put into the separating funnel. 80ml of nitric-acid yttrium solution of 1.5 mol/l was added here, and it sealed, shaked with the shaker, and considered as the w/o emulsion. This was agitated so that between phase splitting might not be carried out to the next reaction. Apart from this, it is kerosine. After dissolved sorbitan monooleate 1.5g in 200ml, putting this into the separating funnel, sealing by having added 40ml of aqueous ammonia, and 20ml of pure water 28%, shaking with a shaker and considering as an w/o emulsion, it moved to the 1l. beaker and agitated by the impeller attached in the motor. The emulsion of the above-mentioned nitric-acid yttrium was previously poured here in 30 seconds. After performing churning for 30 more minutes, it carried out the ** exception with the Buchner funnel. precipitate for the magnetism crucible after 500ml water washes -- the inside of an electric furnace -- the bottom of air atmosphere a temperature up is carried out over 1 hour to 900 degrees C -- it cooled radiationally, after maintaining at 900 degree C for 1 hour 13. The yttrium oxide of 4 g was obtained. The mean particle diameter was 22.5 micrometers, and when the angle of repose was observed with the electron microscope 39 degrees, all almost consisted of a spherical particle.

[0016] It is 40ml of 28% aqueous ammonia about the emulsion of the nitric-acid yttrium solution which did not carry out making example 2 aqueous ammonia react by making it an emulsion, but was generated like the example 1. Into the solution which mixed and obtained 460ml pure water, it poured in in 30 seconds, agitating. it is made below to be the same as that of an example 1 -- the yttrium oxide of 13.3 g was obtained The mean particle diameter was 14.7 micrometers, and when the angle of repose was observed with the electron microscope 41 degrees, all almost consisted of a spherical particle.

[0017] everything but using the nitric-acid gadolinium solution of 1.5 mol/l instead of example 3 nitric-acid yttrium solution is made to be the same as that of an example 1 -- the oxidization gadolinium of 21.5 g was obtained The mean particle diameter was 30.8 micrometers, and when the angle of repose was observed with the electron microscope 36 degrees, all almost consisted of a spherical particle.

[0018] everything but using the nitric-acid neodymium solution of 1.5 mol/l instead of example 4 nitric-acid yttrium solution is made to be the same as that of an example 1 -- the neodymium oxide of 19.8 g was obtained The mean particle diameter was 25.8 micrometers, and when the angle of repose was observed with the electron microscope 38 degrees, all almost consisted of a spherical particle.

[0019] instead of adding 40ml of 528% aqueous ammonia of examples, and 20ml of pure water -- sodium-hydroxide solution of 10 mol/l everything but using 180ml is made to be the same as that of an example 1 -- the yttrium oxide of 13.3 g was obtained The mean particle diameter was 19.6 micrometers, and when the angle of repose was observed with the electron microscope 40 degrees, all almost consisted of a spherical particle.

[0020] 40ml of 128% aqueous ammonia of examples of comparison It is the nitric-acid yttrium solution of 0.75 mol/l, agitating in the solution which mixed and obtained 460ml pure water. It poured in having bet [160] it for 3 minutes. hereafter, it is made to be the same as that of an example 1 -- the yttrium oxide of 13.5 g was obtained 51.4 micrometers and an angle of repose have very bad flow nature, and a mean particle diameter cannot be measured. When observed with the electron microscope, the 1-several micrometers fine particle was also seen besides the particle of the square indeterminate form.

[0021] instead of [of 228% aqueous ammonia of examples of comparison] -- oxalic acid dihydrate everything but adding 25g is made to be the same as that of an example 2 -- the yttrium oxide of 13.3 g was obtained Mean particle diameter Flow nature cannot be measured very bad by 5.4 micrometers and the angle of repose. When observed with the electron microscope, it consisted of a particle of the square indeterminate form. Moreover, when some precipitate before baking was taken and the X-ray diffraction pattern was measured, it was the usual oxalic acid yttrium.

[0022]

[Effect of the Invention] The spherical particle of a rare-earth-elements oxide can be easily obtained by this invention, all almost consist of a spherical particle, it covers also with an angle of repose at the time of the powder for ****, and baking of a ceramic sintered compact small, and this is useful as an object for powder again.

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CLAIMS

[Claim(s)]
[Claim 1] The manufacture method of the rare-earth-elements hydroxide characterized by making it distribute in the organic solvent which is not mixed with water and homogeneity, and adding at least one of this rare-earth-elements solution and these alkaline-water solutions in case mix, the solution of the water-soluble salt of rare earth elements and the solution of ammonia or hydroxylation alkali are made to react and precipitation of a rare-earth-elements hydroxide is obtained.
[Claim 2] The manufacture method of the rare-earth-elements oxide characterized by calcinating the rare-earth-elements hydroxide obtained by the method according to claim 1.

[Translation done.]

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TITLE: PRODUCTION OF HYDROXIDE OF RARE EARTH ELEMENT AND
PRODUCTION OF OXIDE OF RARE EARTH ELEMENT

PUBN-DATE: May 14, 1996

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ABSTRACT:

PURPOSE: To easily produce spherical particles of oxide of a rare earth element useful as powder for thermal spraying and bedding powder spread at the
time of sintering a ceramic compact.

CONSTITUTION: When an aq. soln. of a water-soluble salt of a rare earth element is mixed with an aq. soln. of ammonia or an alkali hydroxide and they are brought into a reaction to form a precipitate of hydroxide of the rare earth element, at least one of the aq. solns. is dispersed in an org. solvent

which is not uniformly miscible with water before the mixing and the resultant precipitate is fired.

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(54)【発明の名称】　希土類元素水酸化物の製造方法及び希土類元素酸化物の製造方法

(57)【要約】　（修正有）

【目的】　熔射用粉末やセラミック焼結体焼結時の敷き粉として有用な希土類元素酸化物の球状粒子を容易に製造する方法。

【構成】　希土類元素の水溶性塩の水溶液と、アンモニアまたは水酸化アルカリの水溶液とを混合して反応させて希土類元素水酸化物の沈殿を得る際に該希土類元素水溶液と該アルカリ水溶液のうちの少なくとも一方を水と均一に混じり合わない有機溶剤中に分散させて加えることを特徴とする希土類元素水酸化物の製造方法、およびそれを焼成することによる希土類元素酸化物の製造方法。

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【特許請求の範囲】

【請求項１】　希土類元素の水溶性塩の水溶液と、アンモニアまたは水酸化アルカリの水溶液とを混合して反応させて希土類元素水酸化物の沈殿を得る際に、該希土類元素水溶液と該アルカリ水溶液のうちの少なくとも一方を水と均一に混じり合わない有機溶剤中に分散させて加えることを特徴とする希土類元素水酸化物の製造方法。

【請求項２】　請求項１に記載の方法で得られた希土類元素水酸化物を焼成することを特徴とする希土類元素酸化物の製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は溶射用粉末、あるいはセラミックス成形焼結体焼成時の敷き粉用として有用な球状希土類元素酸化物の製造方法に関するものである。

【０００２】

【従来の技術】希土類元素酸化物の溶射膜を生成しようとする場合、バーナーに供給する粉末の流れ性が良い方が均一な膜を得るために都合が良く、したがって球状の粒子が好まれる。また、セラミックス成形体を焼結する際に炉材との付着、反応を防ぐために敷く敷き粉としても、流動性が良く接触面積の少なくて済む球状粒子が好まれる。

【０００３】特開平 3-23214号には微粉状の希土類元素酸化物を用いて造粒して球状粒子を得る方法が開示されているが、この方法では、一度溶液から沈殿生成、沪別、焼成によって微粒子を製造した後でそれを用いて製造するので手間がかかる。

【０００４】

【発明が解決しようとする課題】本発明は上記欠点を解決したもので、溶射用及び敷き粉用に適した希土類元素酸化物球状粒子を水溶液から容易に得られる製造方法を提供しようとするものである。

【０００５】

【課題を解決するための手段】本発明者らは、かかる課題を解決するために検討を重ね、生産性が良く簡便な工程からなる溶射用及び敷き粉用に適した球状希土類元素酸化物の製造方法を見出し、製造条件を確立して本発明を完成させたもので、その要旨は、希土類元素の水溶性塩の水溶液と、アンモニアまたは水酸化アルカリの水溶液とを混合して反応させて希土類元素水酸化物の沈殿を得る際に、該希土類元素水溶液と該アルカリ水溶液のうちの少なくとも一方を水と均一に混じり合わない有機溶剤中に分散させて加えることを特徴とする希土類元素水酸化物の製造方法、およびそれを焼成することによる希土類元素酸化物の製造方法にある。

【０００６】本願発明によると希土類元素の水溶性塩の水溶液から容易に水酸化物が得られ、これを焼成することによって球状粒子状の希土類元素酸化物が簡単に得られ経済的であるとともにこの粒子はほとんど全てが球状

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粒子から成っており、安息角も小さく、溶射用粉末あるいはセラミック焼結時の敷き粉として有用であることが判った。

【０００７】以下、本発明を詳細に説明する。本発明の適応範囲は、希土類元素としてイットリウムおよび原子番号が57～71のランタノイドである。

【０００８】本発明における球状とは真球、及び短径に対する長径の比が 1.5以下の略々球形の粒子を意味する。これは用途上十分な範囲である。またこのような粒子からなる酸化物は、流動性の良さを示す安息角（傾斜法で測定）が通常の酸化物より小さい。平均粒径（D_{50}）は体積基準で表したもので、全粒子体積の50%が平均粒径以下の粒子で占められる。測定法はコールターカウンター（コールター社製商品名）を用いた。

【０００９】本発明の球状希土類元素水酸化物の製造方法としては、希土類元素の水溶性塩の水溶液と、アンモニアまたは水酸化アルカリの水溶液とを反応させて沈殿を析出させる際に、希土類元素水溶液とアルカリ水溶液の少なくとも一方を、水と均一に混じり合わない有機溶剤中に分散させ、有機溶剤の連続相の中に水相が分散したいわゆるw／o型エマルジョンとして加える。希土類元素の水溶性塩としては、硝酸塩、塩化物等が用いられる。希土類元素の種類は１種でも、２種以上でも良く、また全希土類元素濃度はあまり低いと液量が増えて不経済でまた、あまり高いと沈殿生成後に粘度が上がりすぎるので 0.1～2.0mol/lが良い。

【００１０】希土類元素を沈殿させるために水酸化ナトリウム、水酸化カリウム、水酸化バリウム等、またはアンモニアの水溶液を用いるが、アルカリ金属等の混入を嫌う場合には、アンモニア水を用いるのがよい。この濃度も希土類元素溶液の所で述べたのと同じ理由で 0.2～5.0mol/lが良い。量としては、希土類元素１mol に対し３～５mol が適当で、３mol 未満では収率が悪く、５mol を超えても効果はなく不経済である。

【００１１】有機溶剤としては、水と均一に混じり合わないものなら良いが、例として、トルエン等の芳香族炭化水素、ｎ－ヘキサン等の直鎖脂肪族炭化水素、シクロヘキサン等の環状脂肪族炭化水素、ケロシン等の石油の分留によって得られる混合物が挙げられる。中で、引火点の高さ、人体への安全性、価格等を考慮して、ケロシン、シクロヘキサン等が好ましい。量としては、分散させる水溶液に対し、体積で２～５倍の量を用いるのが、w／oエマルジョンの生成に好都合である。

【００１２】また、この際ソルビタンモノオレエート、ポリオキシエチレンソルビタンモノステアレート、ソルビタントリオレエート等の、比較的油溶性の乳化剤を有機溶剤に対して 0.5～ 5.0重量%加える。

【００１３】溶液の混合方法としては、アルカリ溶液および希土類元素溶液のうち、エマルジョンにして加えるものを先ず乳化剤を溶かした有機溶剤と、振り混ぜ、撹

拌等によって混合して、乳化する。w／oエマルジョンがうまくできたかどうかは、光学顕微鏡で油溶性、或は水溶性の色素を加える等の方法で確認できる。希土類元素水溶液とアルカリ水溶液の両方をエマルジョンにする場合は、エマルジョンの同士の混合順序はどちらでも良いが、片方は水溶液のみで混合する場合は、エマルジョンの方を水溶液に加えるのが良い。この混合に要する時間は任意でよい。溶液を混合する際の温度は室温がよい。低温にしても効果はなく、高温にするのは有機溶剤を使っているので安全上好ましくない。

【００１４】すべての溶液を加え終わったら、反応を完全に進行させる為に10分以上おく。沈殿の生成が終了したら、ブフナー漏斗で沪別し、水洗する。これで球状希土類元素水酸化物が得られた。得られた水酸化物を 600℃以上 1,500℃以下で焼成することにより、球状希土類元素酸化物が得られる。

【００１５】

【実施例】以下、本発明の実施態様を実施例を挙げて説明するが、本発明はこれらに限定されるものではない。

実施例１

ケロシン 200mlにソルビタンモノオレエート1.5gを加えて溶解したものを分液漏斗に入れた。ここに、1.5mol/lの硝酸イットリウム水溶液80mlを加え、密栓をして振とう機で振り混ぜ、w／oエマルジョンとした。これは、次の反応までの間分相しないように撹拌しておいた。これとは別に、ケロシン 200mlにソルビタンモノオレエート1.5gを溶解し、これを分液漏斗に入れ、28%アンモニア水40mlと純水20mlを加えて、密栓をして振とう機で振り混ぜてw／oエマルジョンとしたのち、１リットルビーカーに移し、モーターに取り付けた撹拌翼で撹拌した。ここに、先に前述の硝酸イットリウムのエマルジョンを30秒間で注ぎ込んだ。さらに30分間撹拌を行った後、ブフナー漏斗で沪別した。沈殿物を 500mlの水で洗浄したのち、磁性坩堝にとり、電気炉中で大気雰囲気下 900℃まで１時間かけて昇温し、 900℃に１時間保った後放冷した。13.4gの酸化イットリウムが得られた。平均粒径は22.5μmであり、安息角は39度、電子顕微鏡で観察したところ、ほとんど全てが球状粒子からなっていた。

【００１６】実施例２

アンモニア水をエマルジョンにして反応させることをせず、実施例１と同様にして生成した硝酸イットリウム水溶液のエマルジョンを、28%アンモニア水40mlと 460mlの純水を混合して得た水溶液中に、撹拌しながら、30秒間で注ぎ込んだ。以下実施例１と同様にして、 13.3gの

酸化イットリウムを得た。平均粒径は14.7μmであり、安息角は41度、電子顕微鏡で観察したところ、ほとんど全てが球状粒子からなっていた。

【００１７】実施例３

硝酸イットリウム水溶液の代わりに1.5mol/lの硝酸ガドリニウム水溶液を用いることの他は実施例１と同様にして、 21.5gの酸化ガドリニウムを得た。平均粒径は30.8μmであり、安息角は36度、電子顕微鏡で観察したところ、ほとんど全てが球状粒子からなっていた。

【００１８】実施例４

硝酸イットリウム水溶液の代わりに1.5mol/lの硝酸ネオジム水溶液を用いることの他は実施例１と同様にして、 19.8gの酸化ネオジムを得た。平均粒径は25.8μmであり、安息角は38度、電子顕微鏡で観察したところ、ほとんど全てが球状粒子からなっていた。

【００１９】実施例５

28%アンモニア水40mlと純水20mlを加える代わりに、 10mol/lの水酸化ナトリウム水溶液 180mlを用いることの他は実施例１と同様にして、 13.3gの酸化イットリウムを得た。平均粒径は19.6μmであり、安息角は40度、電子顕微鏡で観察したところ、ほとんどすべてが球状粒子からなっていた。

【００２０】比較例１

28%アンモニア水40mlと 460mlの純水を混合して得た水溶液中に、撹拌しながら、 0.75mol/lの硝酸イットリウム水溶液 160mlを３分間かけて注ぎ込んだ。以下、実施例１と同様にして、 13.5gの酸化イットリウムを得た。平均粒径は51.4μm、安息角は流れ性が極めて悪く測定不能。電子顕微鏡で観察したところ、角張った不定形の粒子の他に、１～数μmの細かい粒子も見られた。

【００２１】比較例２

28%アンモニア水の代わりに、蓚酸二水和物 25gを加えることの他は実施例２と同様にして、 13.3gの酸化イットリウムを得た。平均粒径は 5.4μm、安息角は流れ性が極めて悪く測定不能。電子顕微鏡で観察したところ、角張った不定形の粒子からなっていた。また、焼成前の沈殿物の一部をとり、X線回析パターンを測定したところ、通常の蓚酸イットリウムであった。

【００２２】

【発明の効果】本発明により希土類元素酸化物の球状粒子を容易に得ることが出来、これは又、ほとんど全てが球状粒子からなっており、安息角も小さく熔射用粉末や、セラミック焼結体の焼成時の敷き粉用として有用なものである。